

Microstructural effects and kinetics of high temperature oxidation in Nb-Si base alloys

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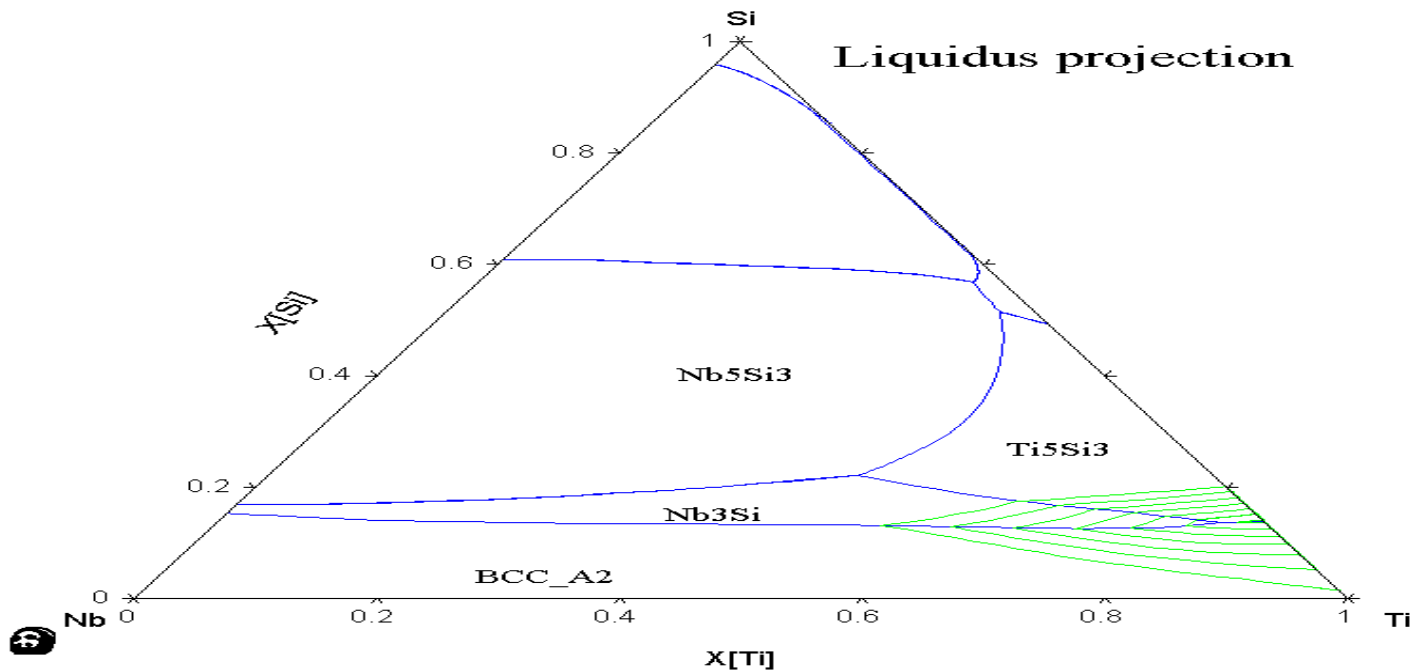
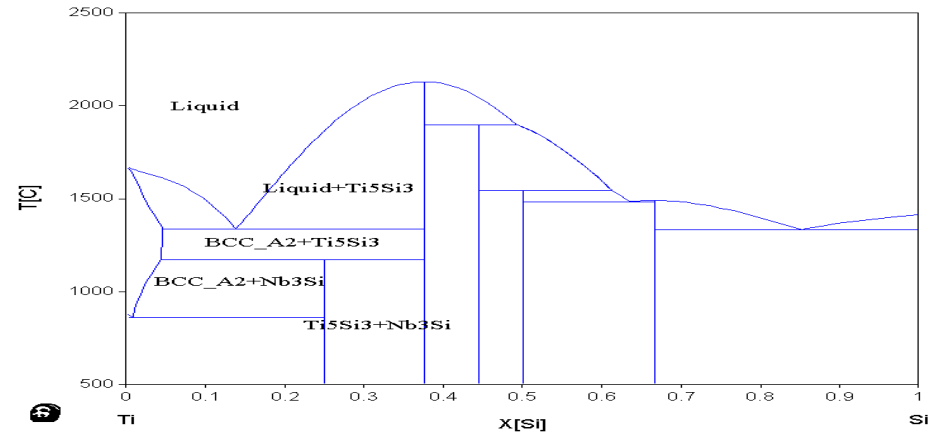
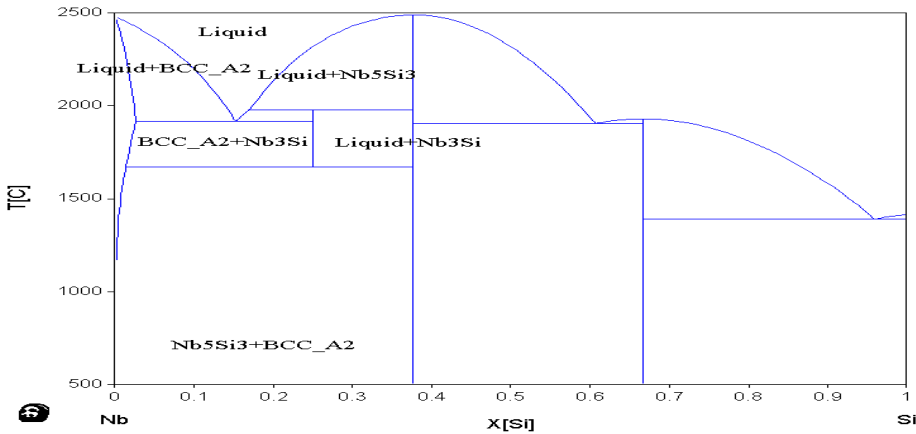


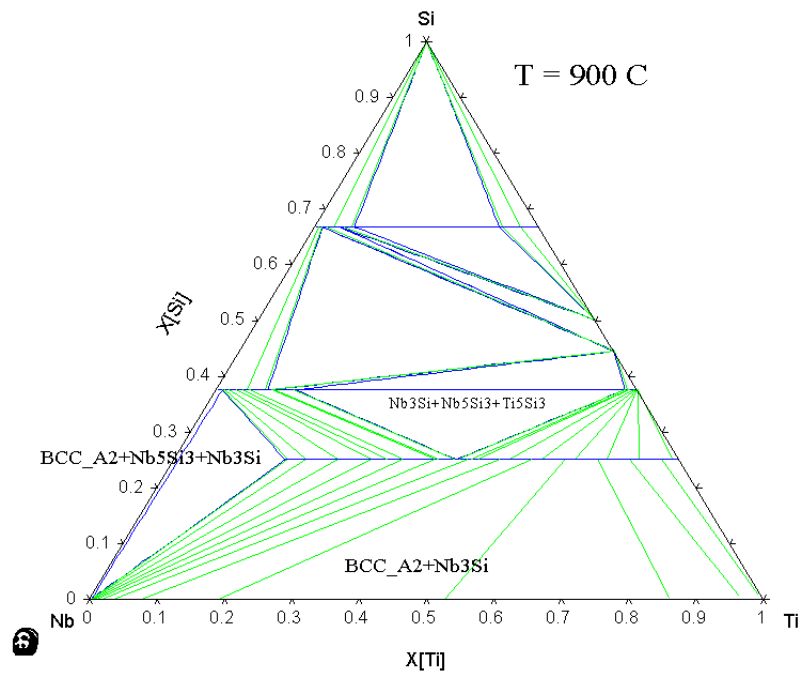
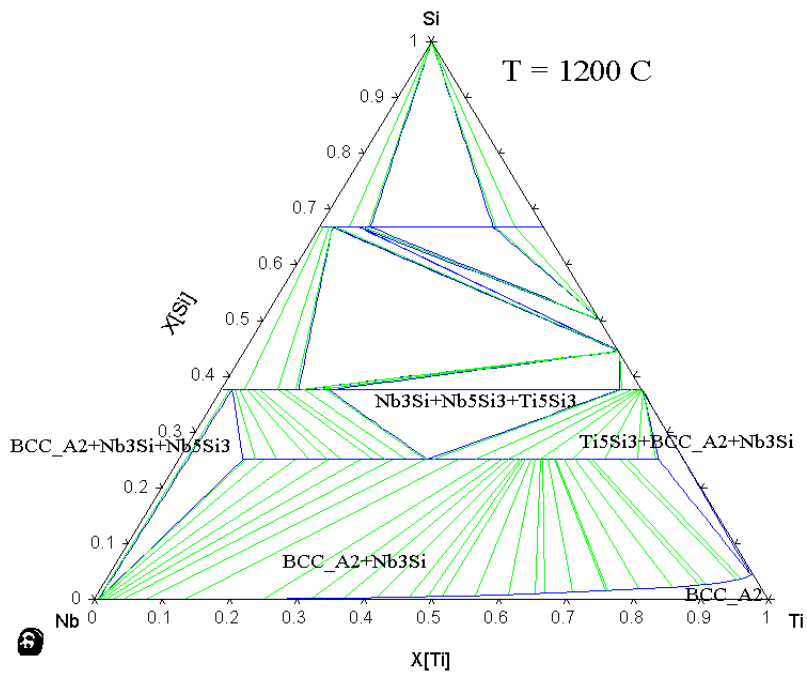
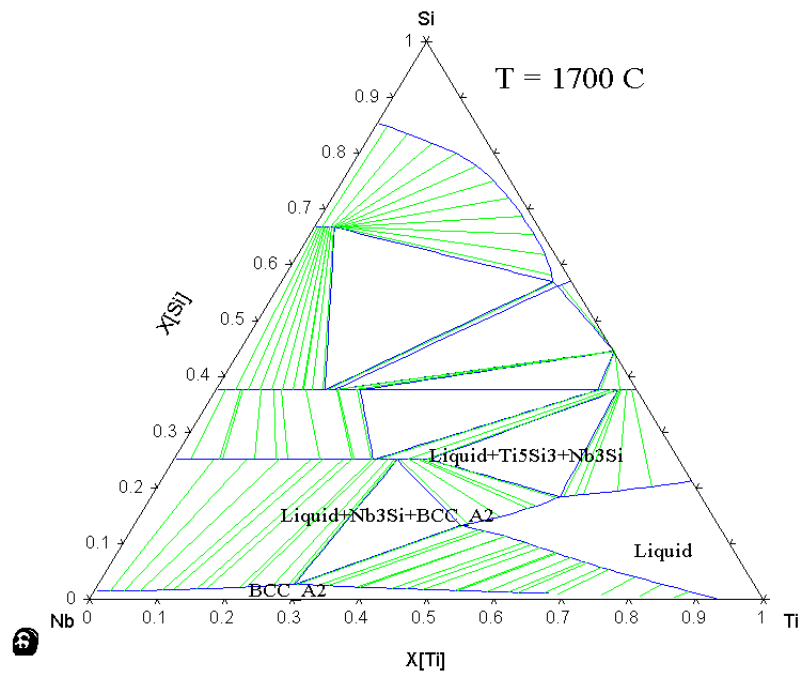
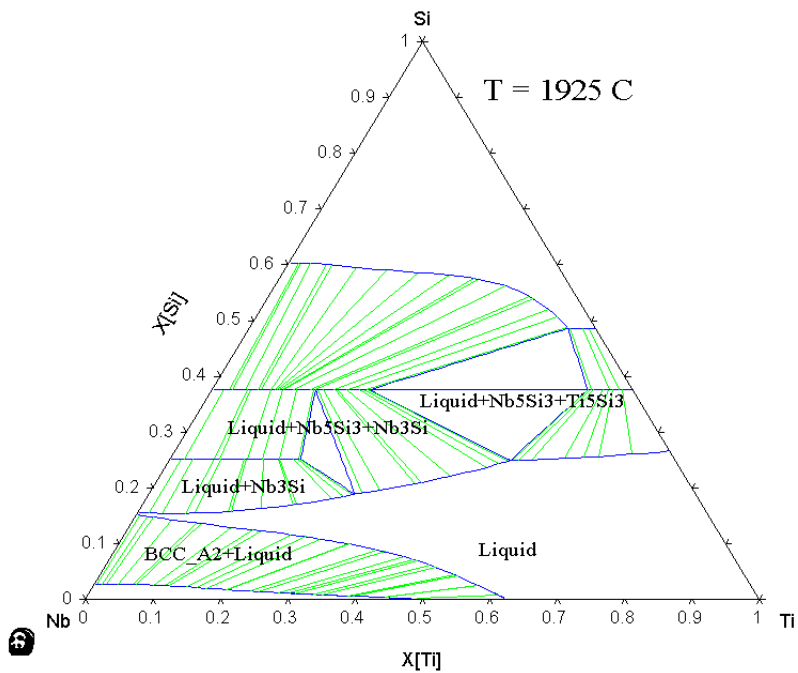
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INTRODUCTION

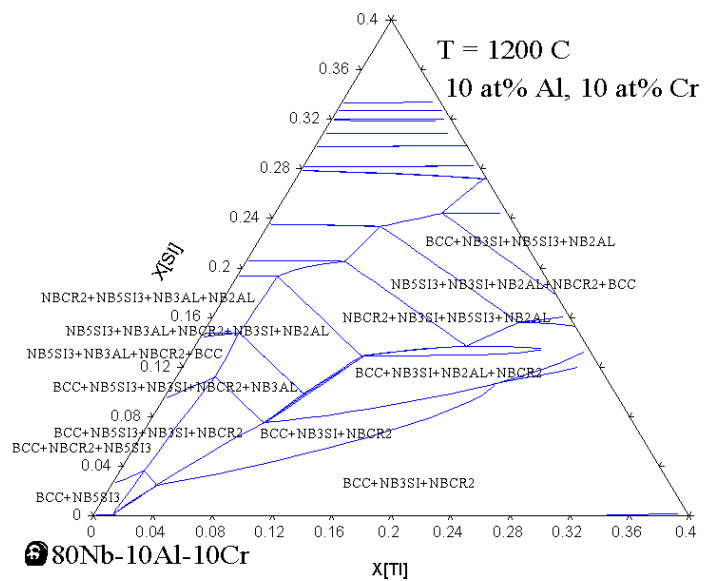
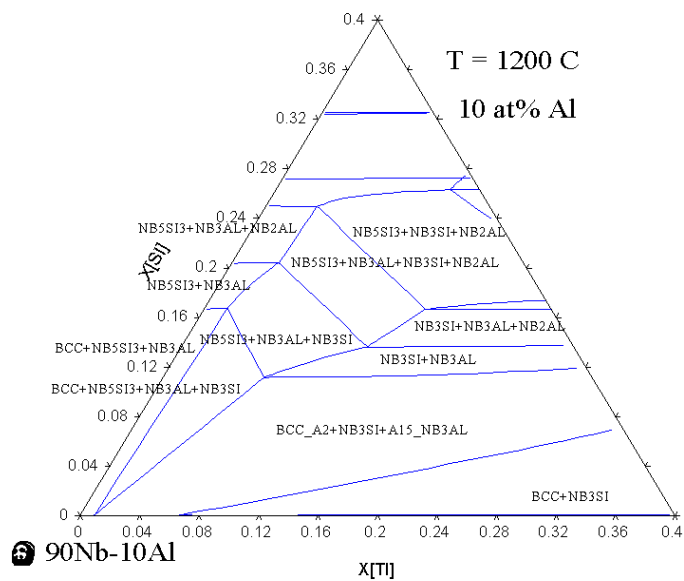
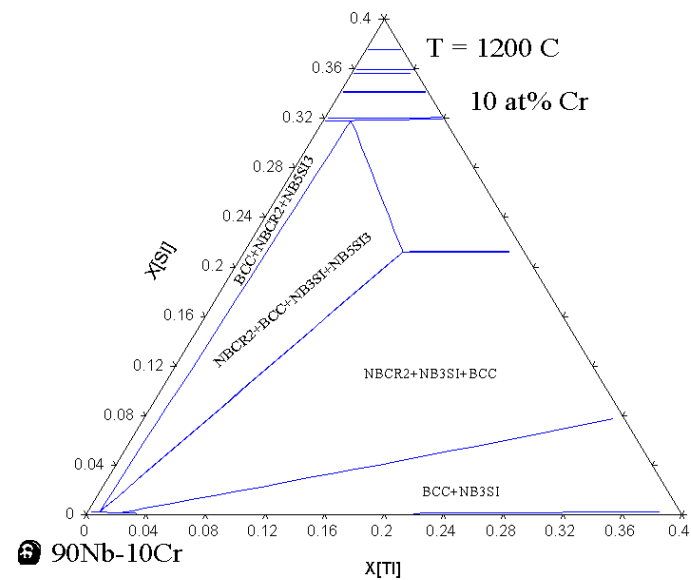
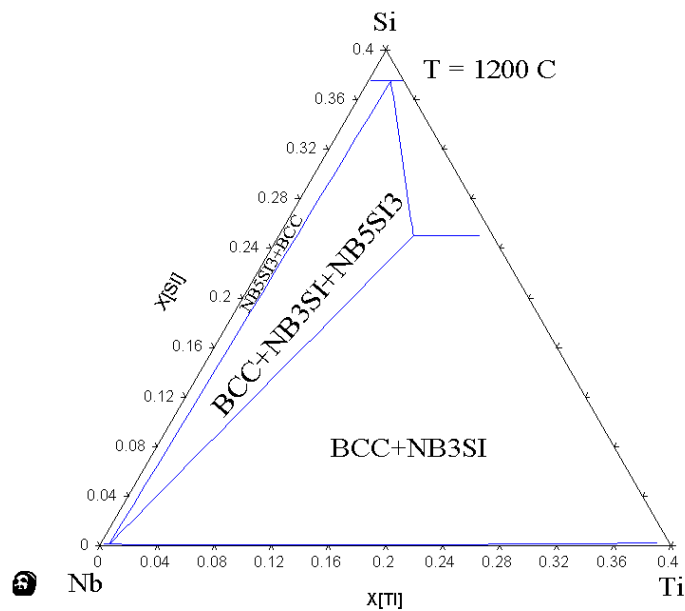
- **Understand the effect of alloying on microstructural modification in Nb-Ti-Si based alloys (phases formed, microstructural distribution)**
- **Effect of Ti, Al, Cr, C on Nb-Si alloys (as-cast, heat-treated alloys)**
- **Oxidation effects on microstructures**
- **Future directions in the study**

Phase diagrams calculated using PANDAT (Y.A. Chang et al. Computherm, LLC)



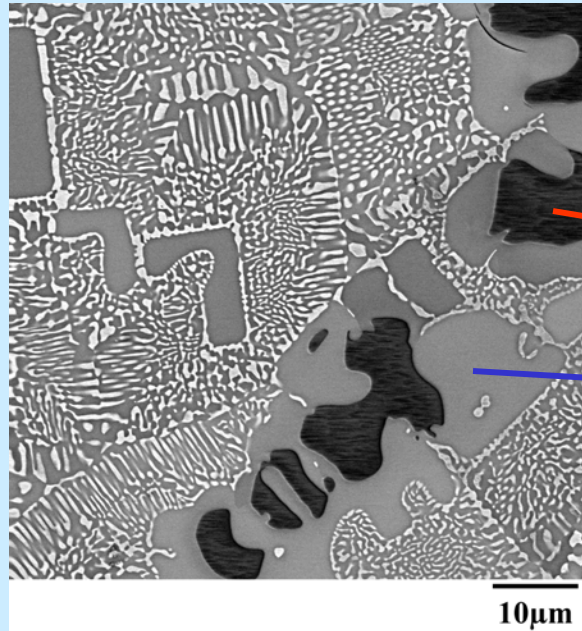


Calculated Isothermal sections at 1200°C

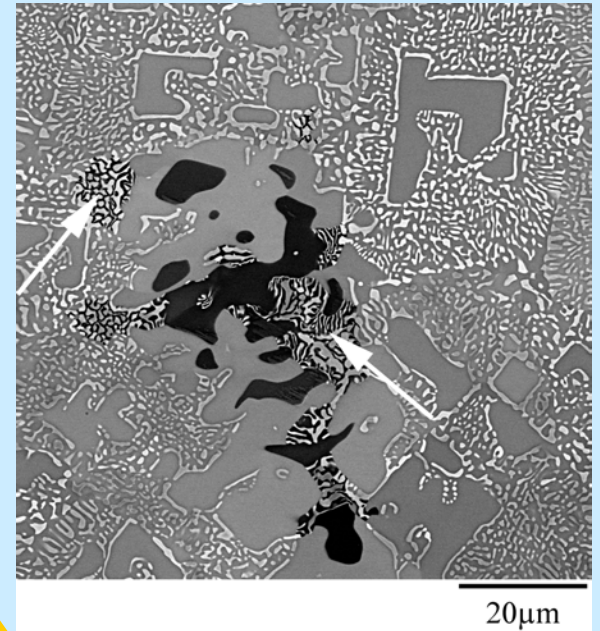


$\text{Nb}_3\text{Si} \rightarrow \beta + \text{Nb}_5\text{Si}_3$ eutectoid reaction

Nb-17.2 at%Si



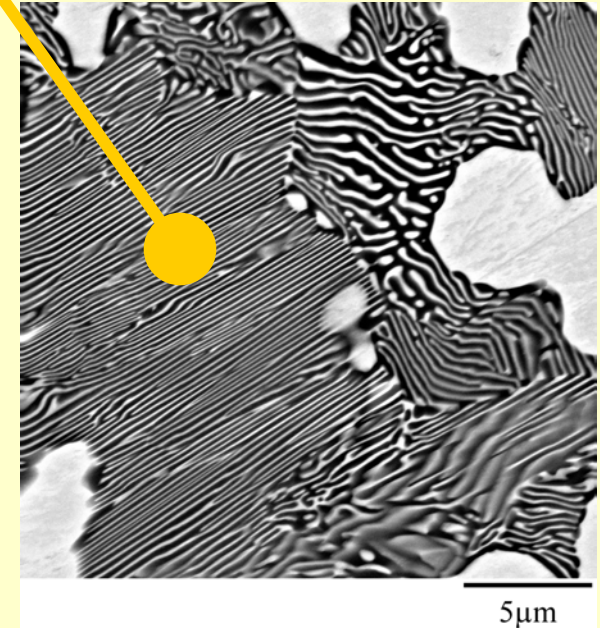
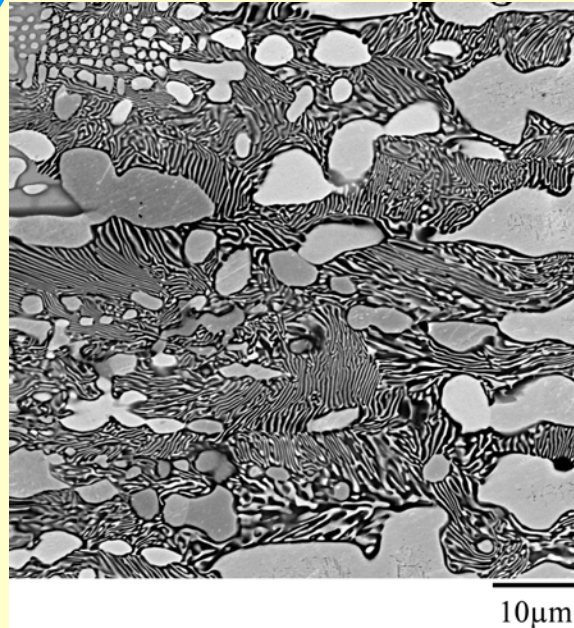
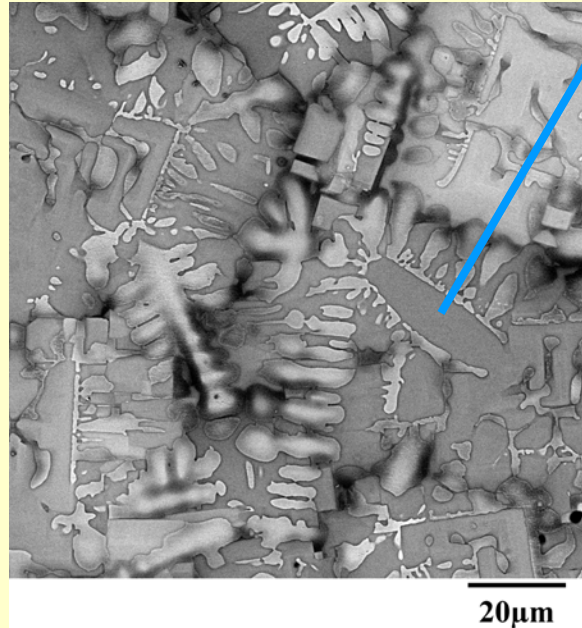
As cast



1200°C – 120 hours

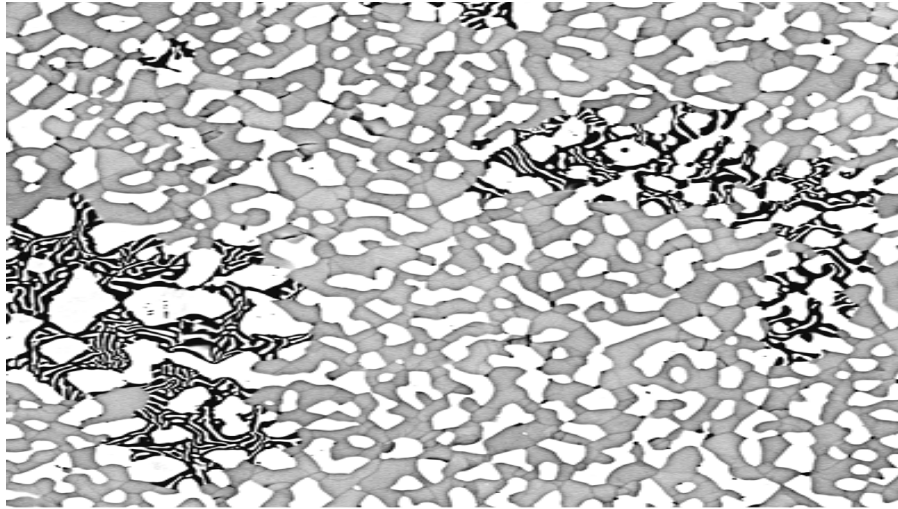
~Nb-22Si-12Ti

Nb-15.0Si-12.0Ti



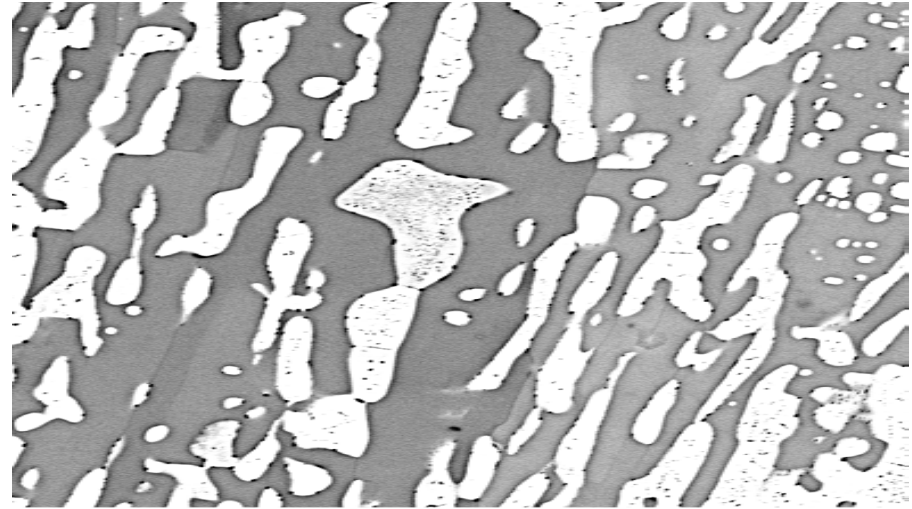
Nb-15.8Ti-16.0Si

1100°C / 185 hrs



10µm

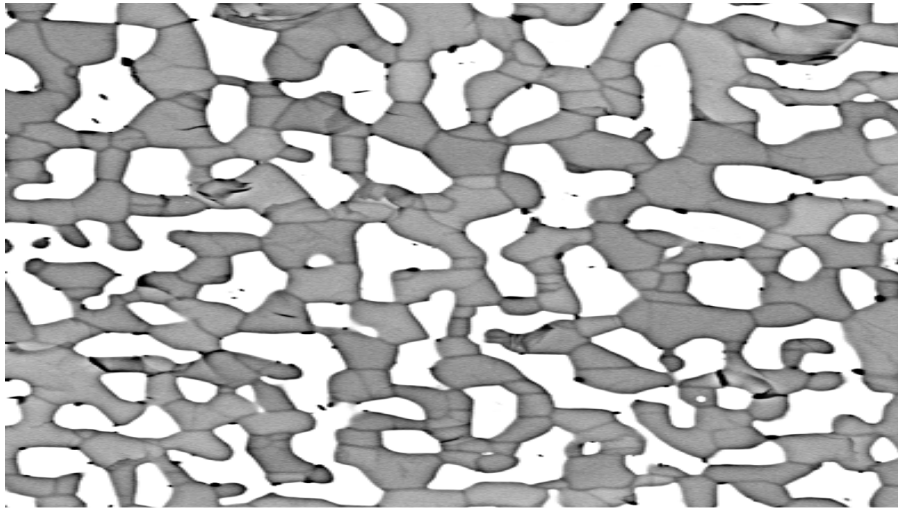
1000°C / 257 hrs



10µm

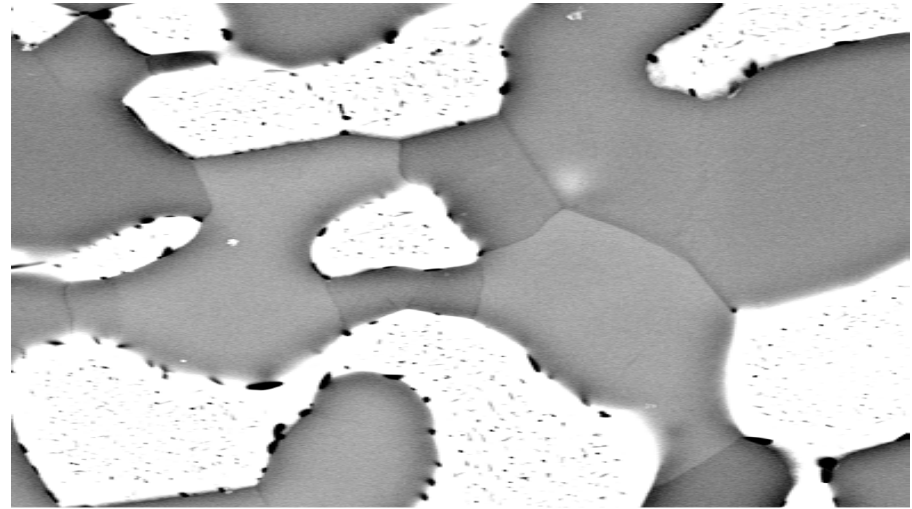
Nb-20.8Ti-15.8Si

1100°C / 185 hrs



10µm

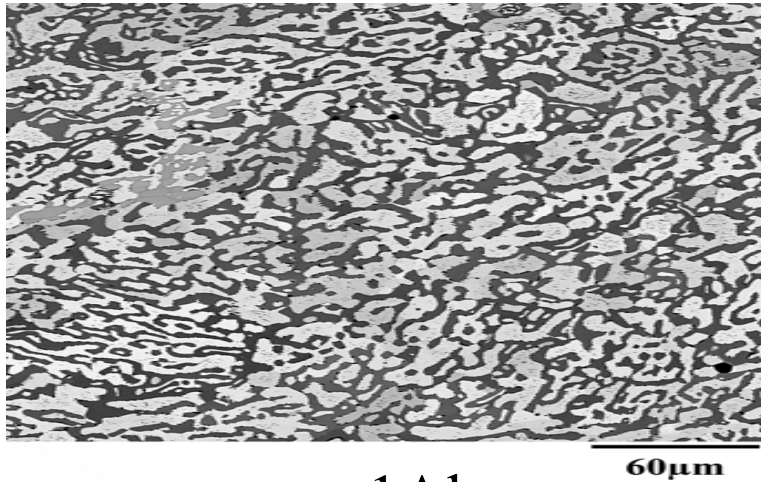
800°C / 576 hrs



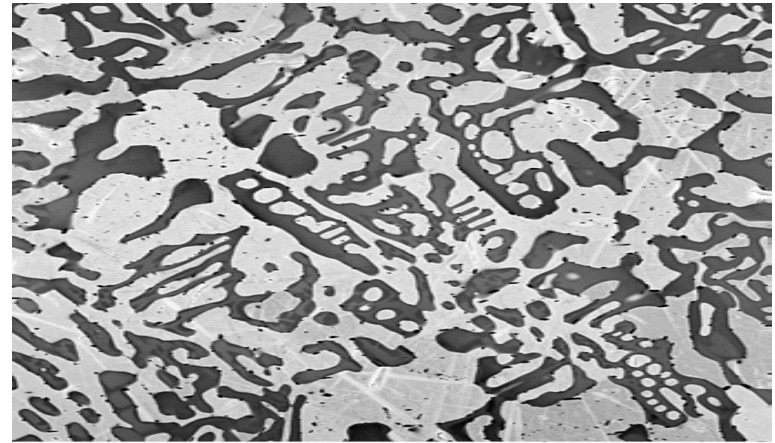
2µm

Nb-16.5Si-20Ti-xAl

1500°C 100 hours



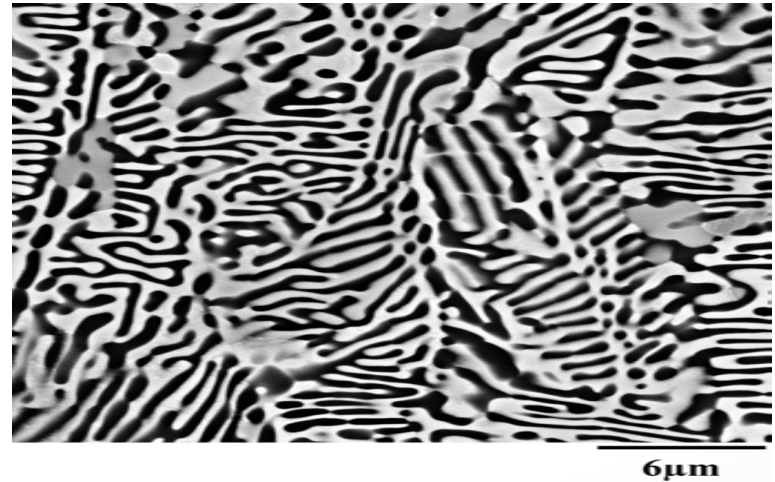
1Al



2.5Al



5Al

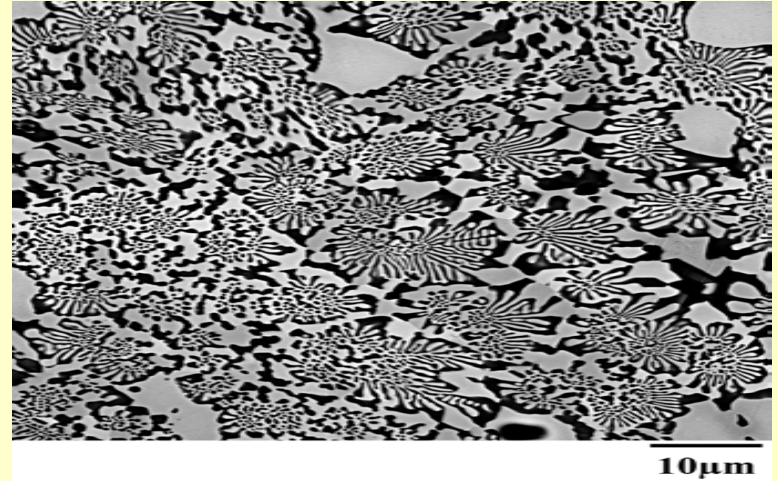
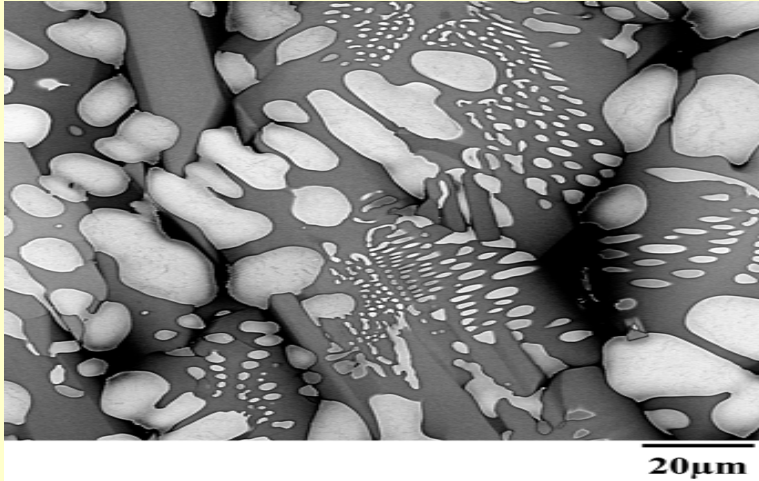


Nb-16.5Si-5Al

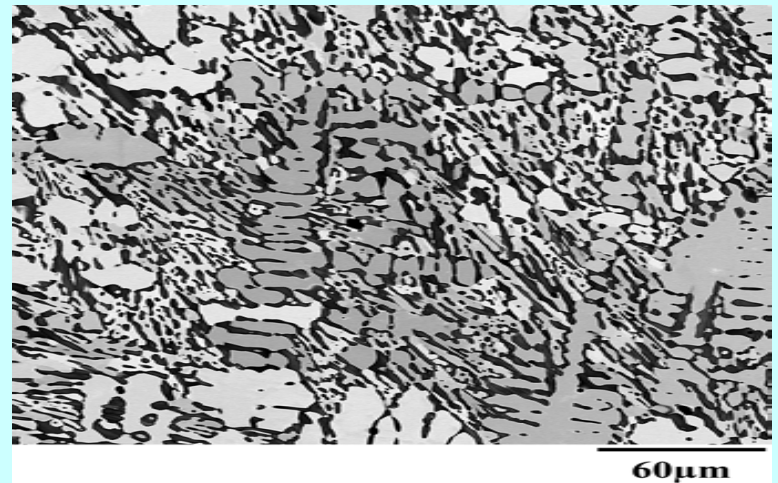
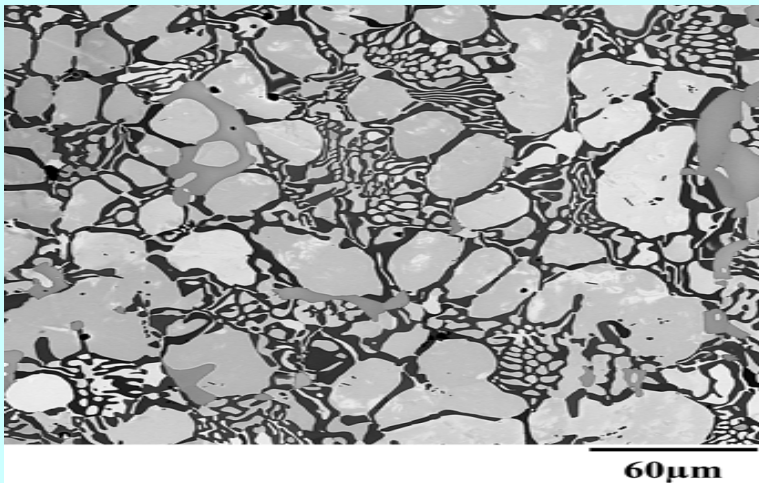
Nb-14Si-12.5Ti

Nb-14Si-12.5Ti-2C

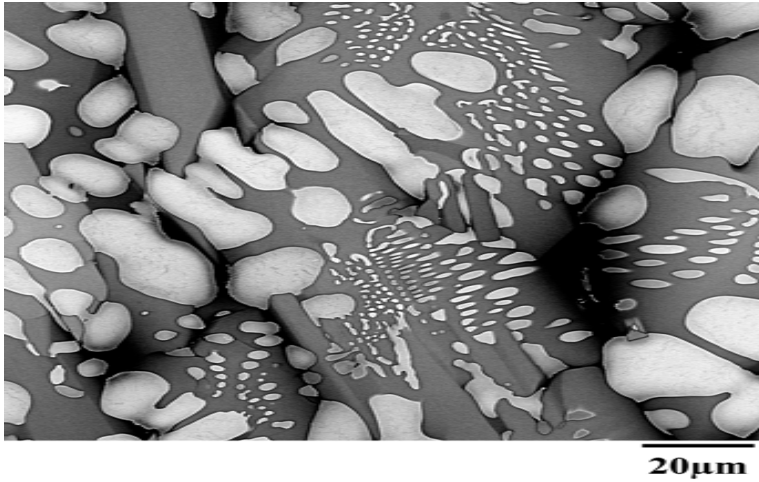
As cast



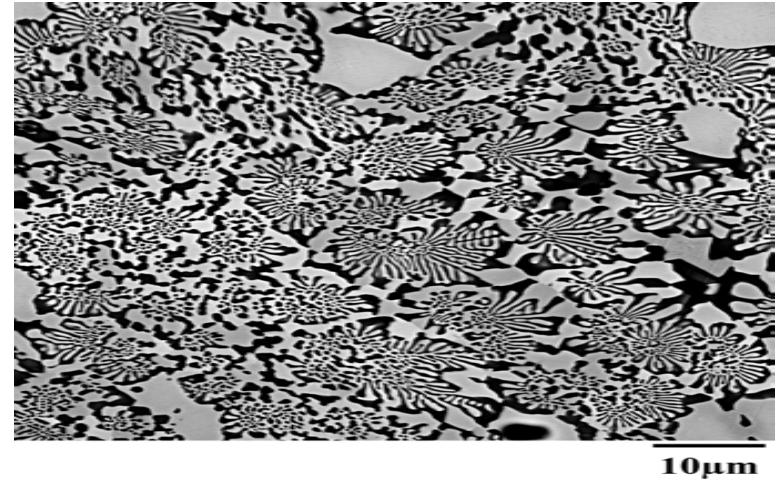
1500°C 100 hours



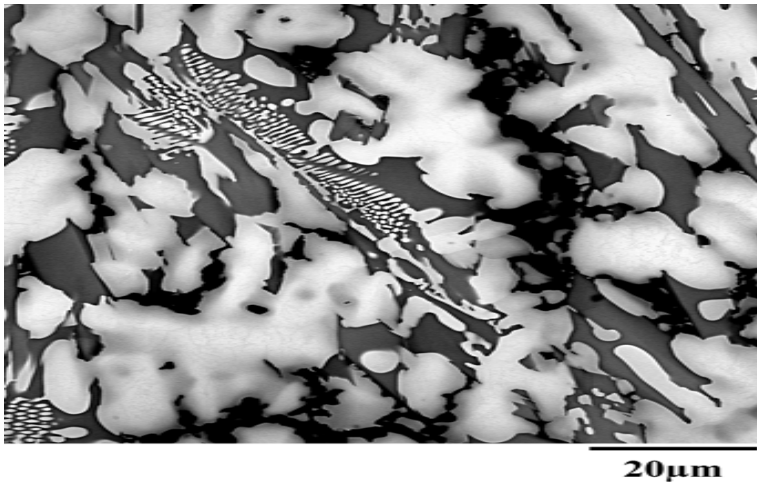
As cast microstructures



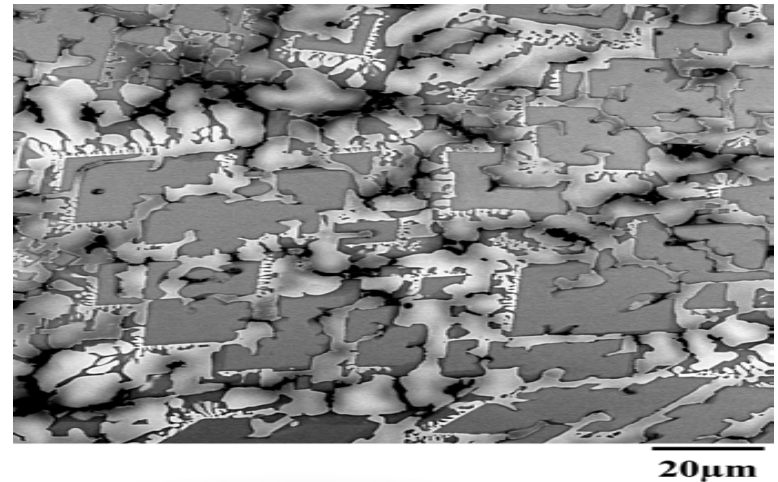
Nb-14Si-12.5Ti



Nb-14Si-12.5Ti-2C



Nb-14Si-12.5Ti-10Cr



Nb-14Si-12.5Ti-10Cr
-10Al

Equilibrium Phases and their Compositions at 1200°C

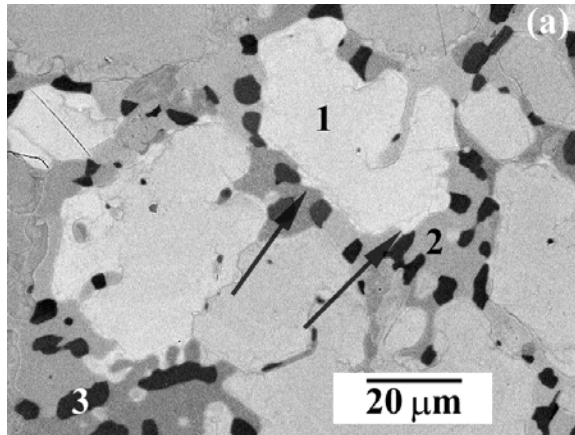


Fig. 1 (a) Nb-19.86Ti-19.74Si-4.21Ge-3.26Al-4.21Hf-9.90Cr

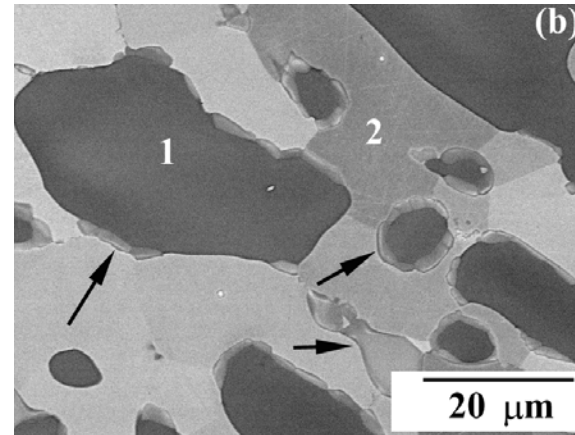
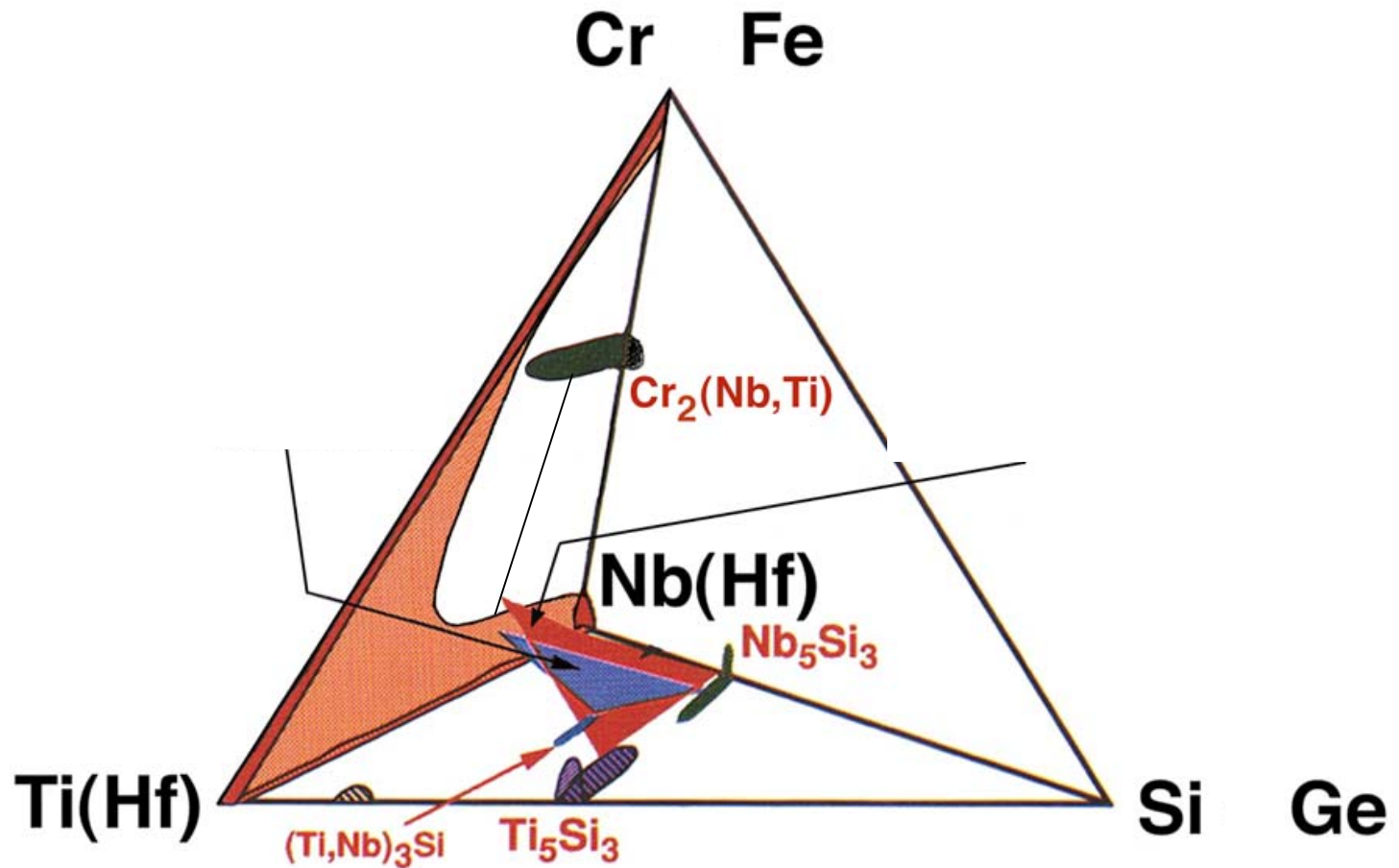


Fig. 1(b) Nb-25.99Ti-12.61Si-4.94Ge-1.92Al-1.90Hf-6.73Cr-0.43Sn.

1 : Nb₅Si₃ ; 2 : β solid solution phase; 3: Cr₂Nb; Arrows : Ti₅Si₃

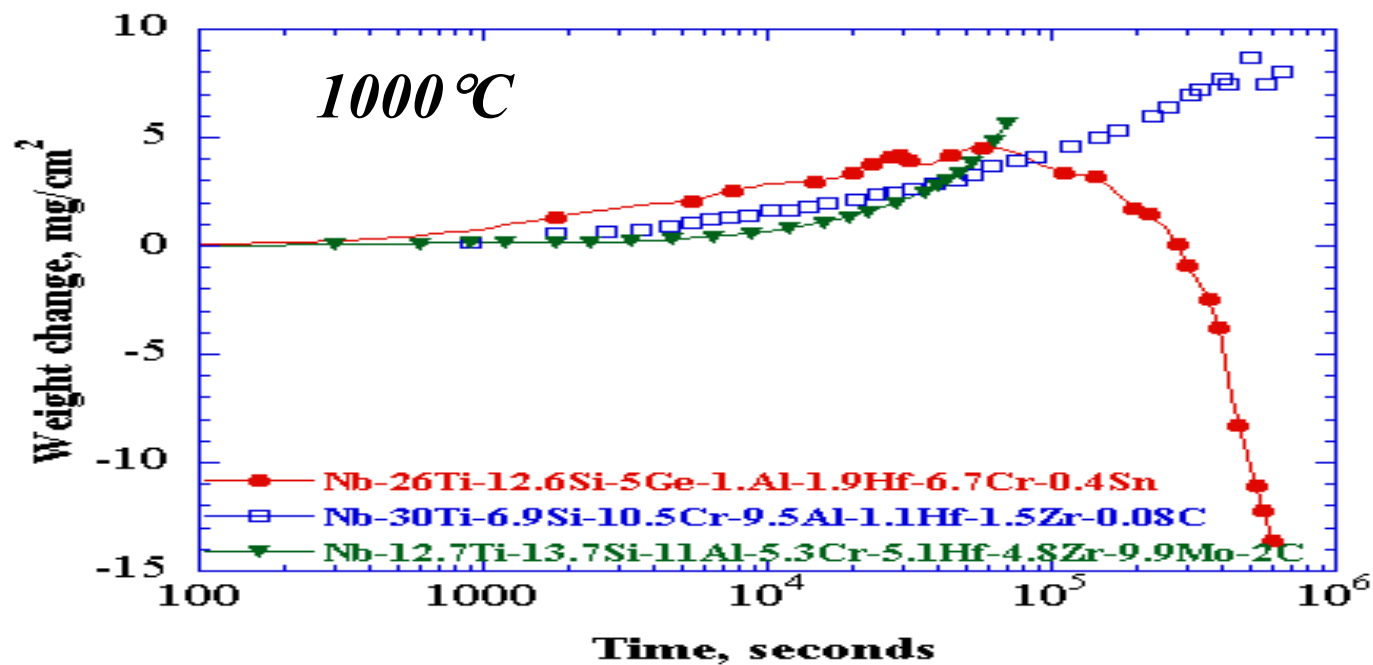
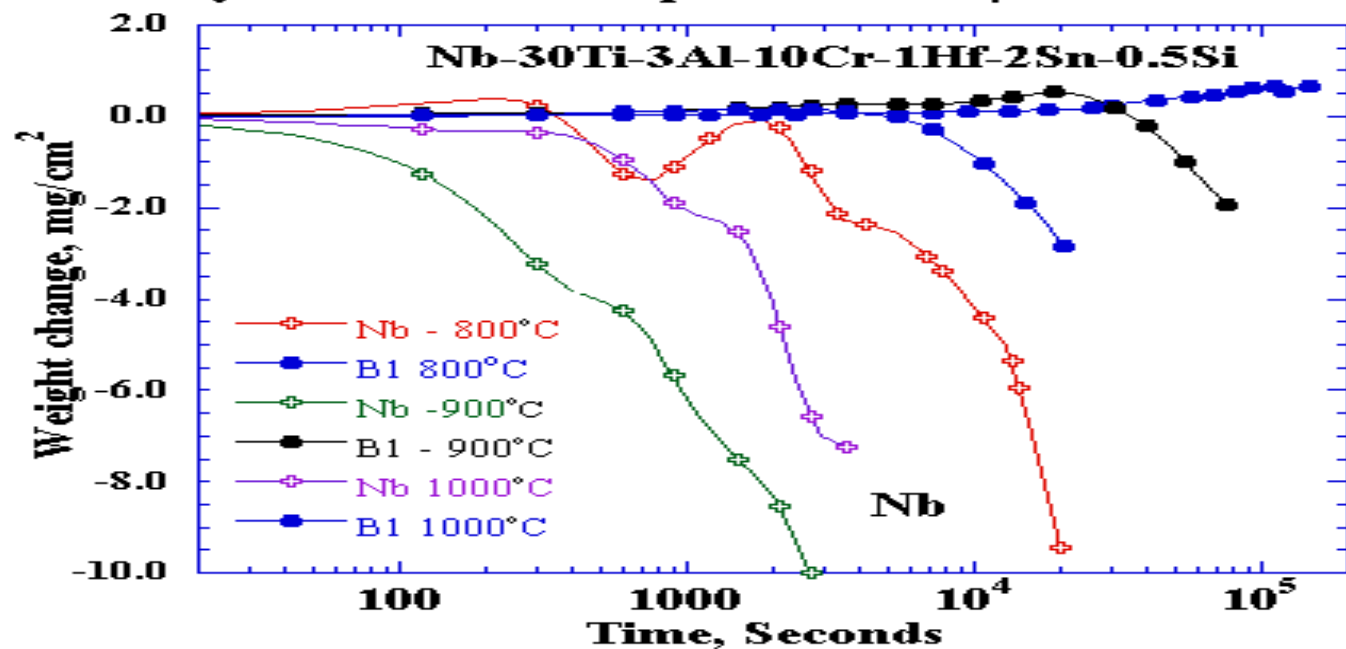
At. %	β		Nb ₅ Si ₃ -type		Ti ₅ Si ₃ -type		Cr ₂ Nb -type
	Fig 1(a)	Fig. 1(b)	Fig. 1(a)	Fig. 1(b)	Fig. 1(a)	Fig. 1(b)	Fig. 1(a)
Nb	53.07	57.99	38.04	38.59	28.28	26.80	21.78
Ti	30.23	26.58	18.04	22.23	26.22	29.18	12.65
Si	0.45	0.50	30.83	25.78	27.67	26.47	6.05
Ge	0.16	0.06	5.72	7.34	7.80	9.19	0.34
Hf	1.03	0.72	4.74	1.71	6.73	6.10	4.74
Al	3.35	2.60	1.19	1.54	2.13	1.22	0.98
Cr	11.72	10.06	1.43	2.41	1.17	0.90	54.81
Sn	-	1.51	-	0.41	-	0.14	-

Schematic Phase Diagram

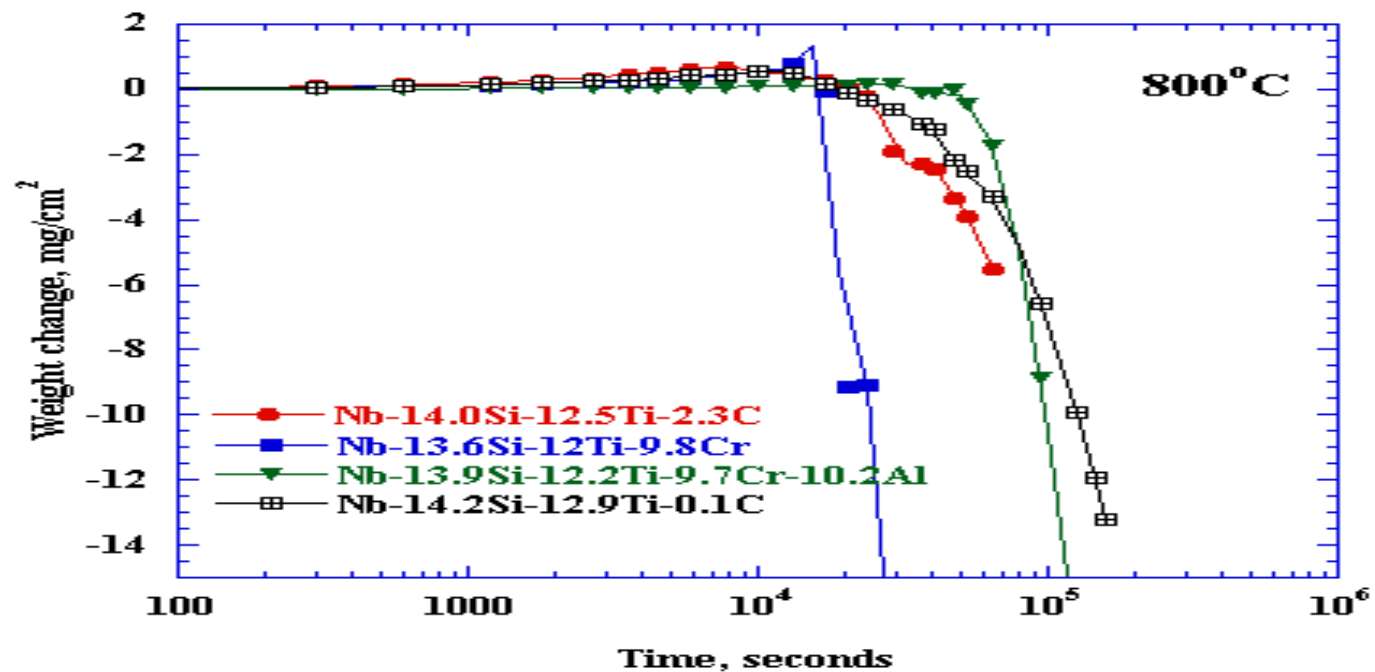
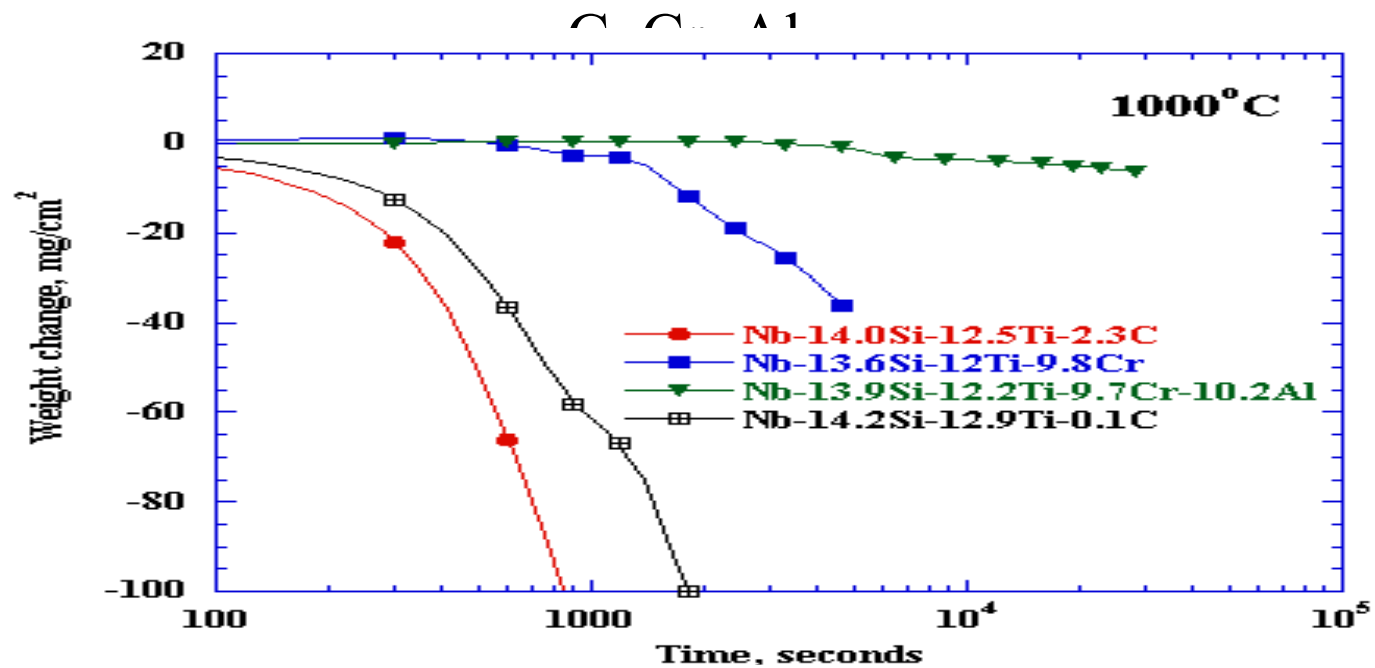


Oxidation resistance of Nb alloys

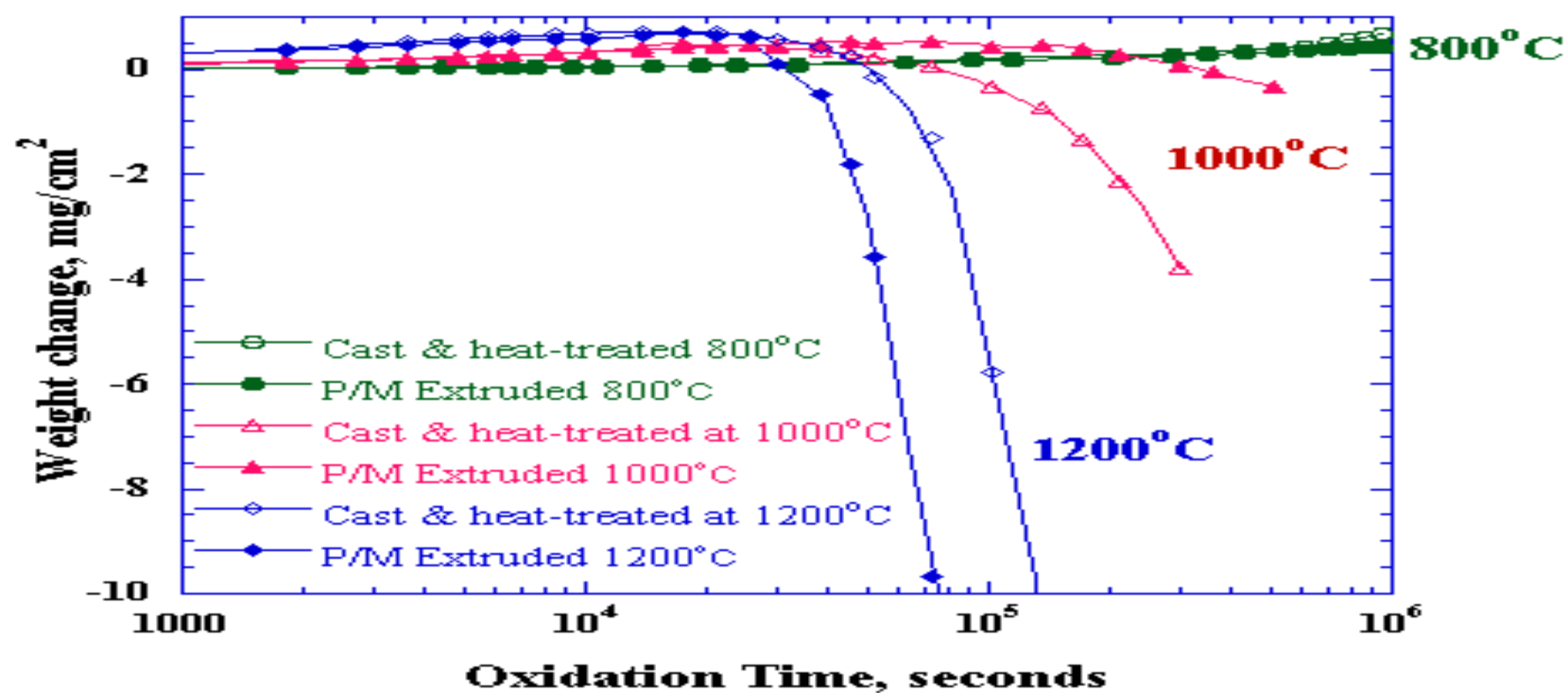
Cyclic oxidation of pure Nb and β solid solution



Effects of alloying on cyclic oxidation

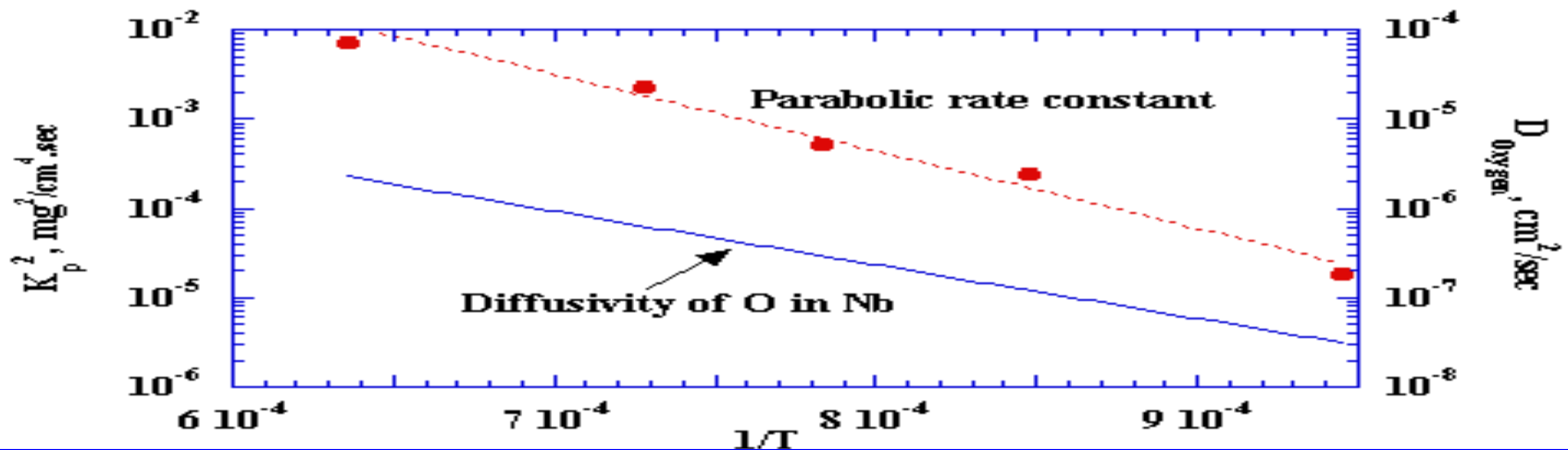
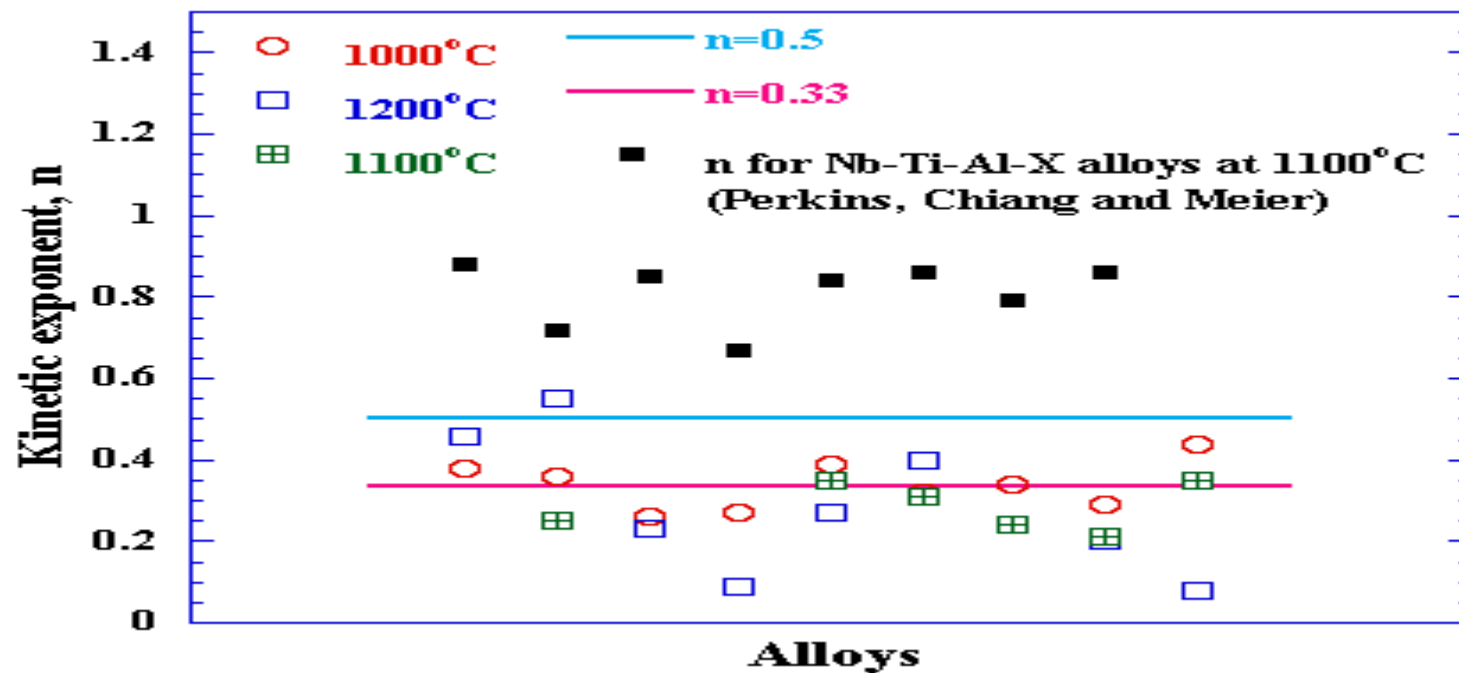


Effect of processing history on cyclic oxidation

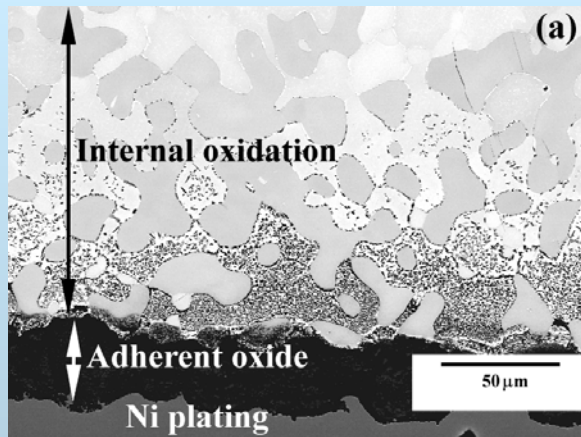


Initial stages of oxidation

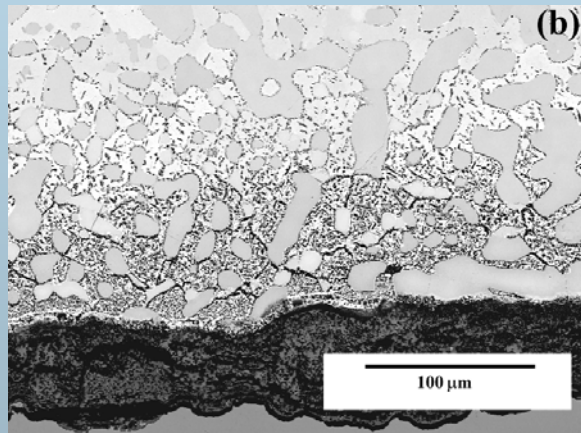
$$\Delta w = Kt^n$$



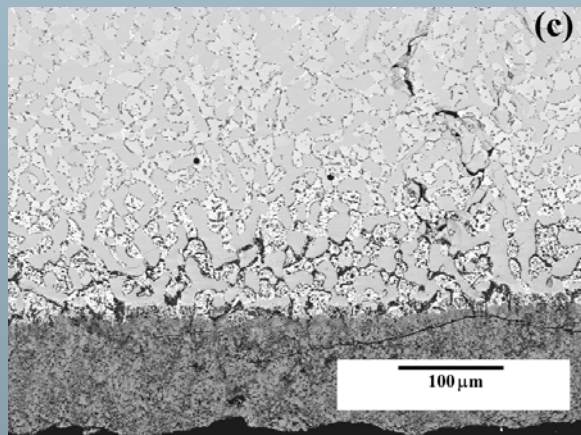
Oxidation of a Nb-26Ti-13Si-5Ge-7Cr-2Al -2Hf-0.5Sn alloy at 1200°C



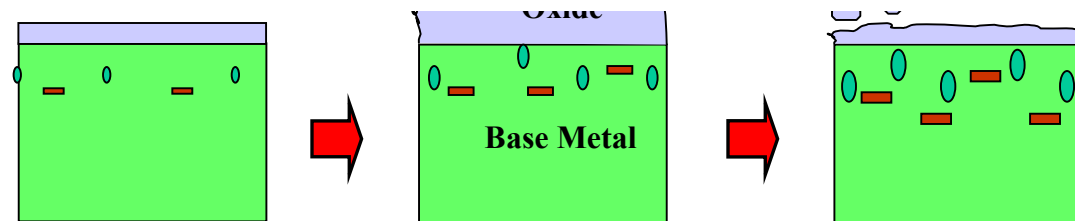
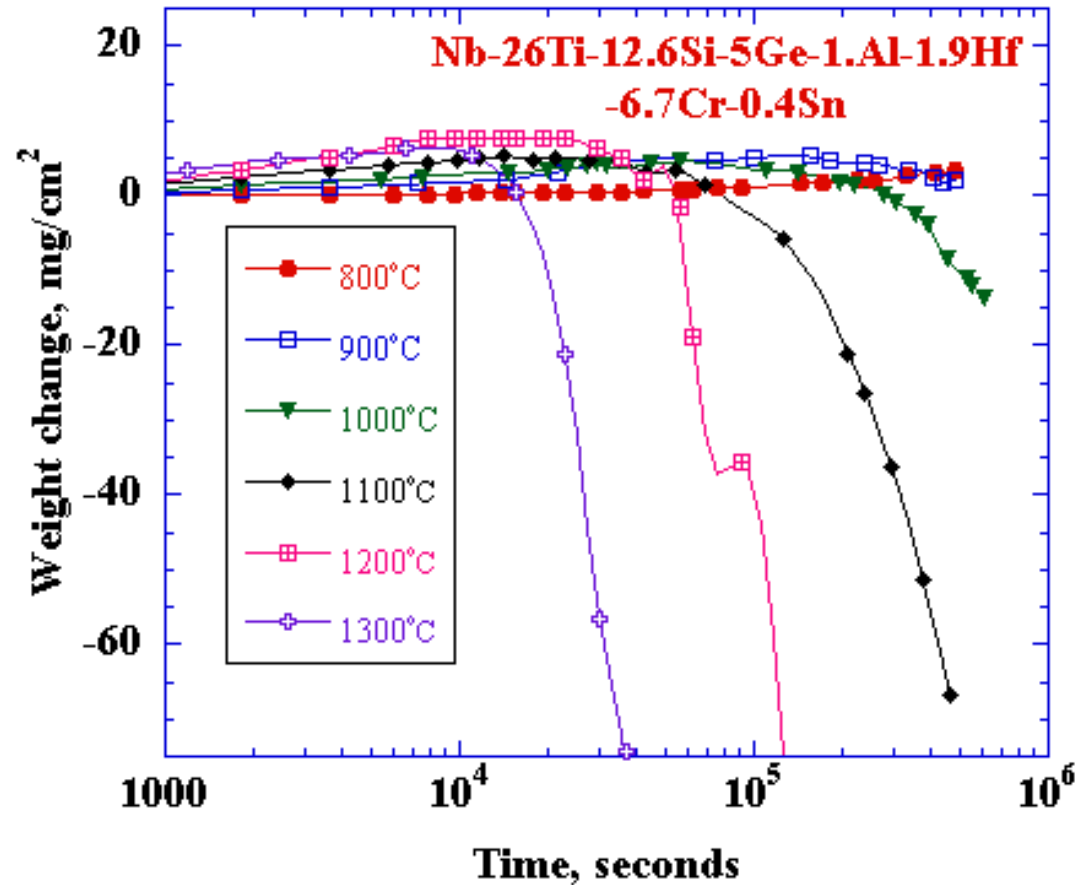
1 hour



5 Hours

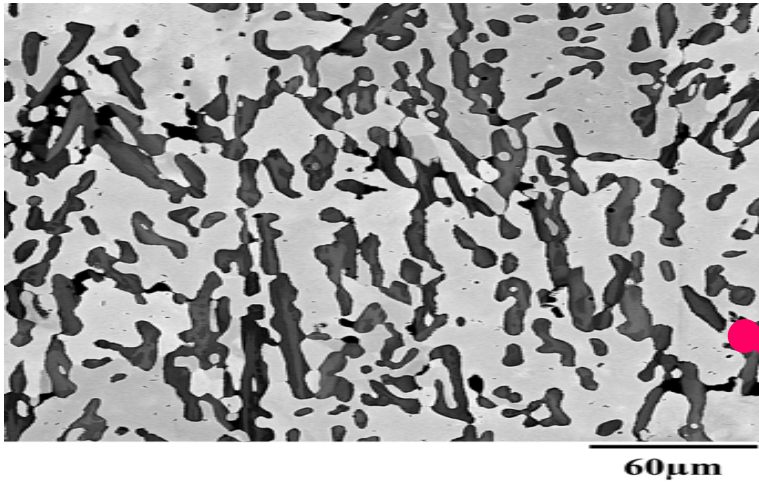


24 hours

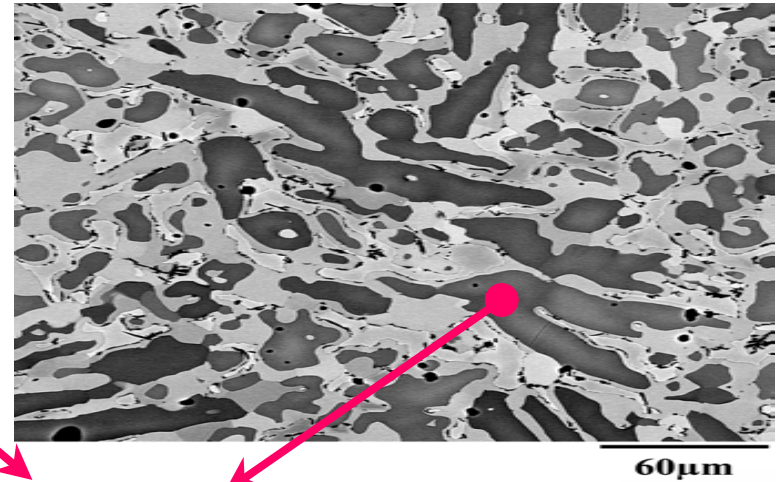


Internal Oxidation

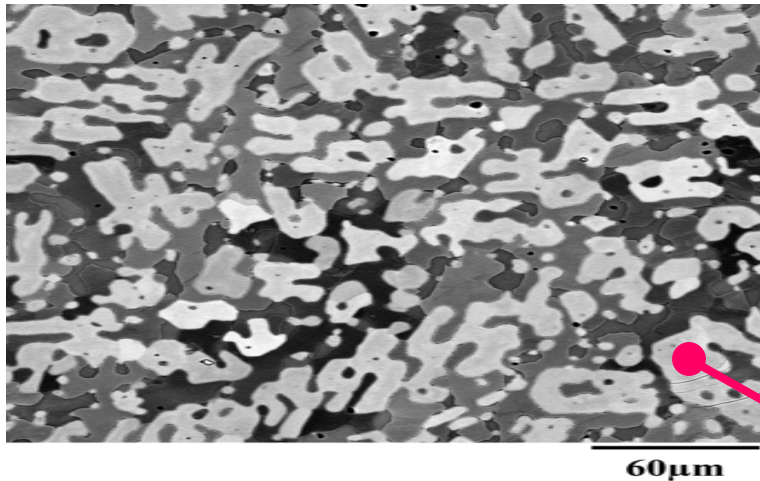
Effect of complex alloying



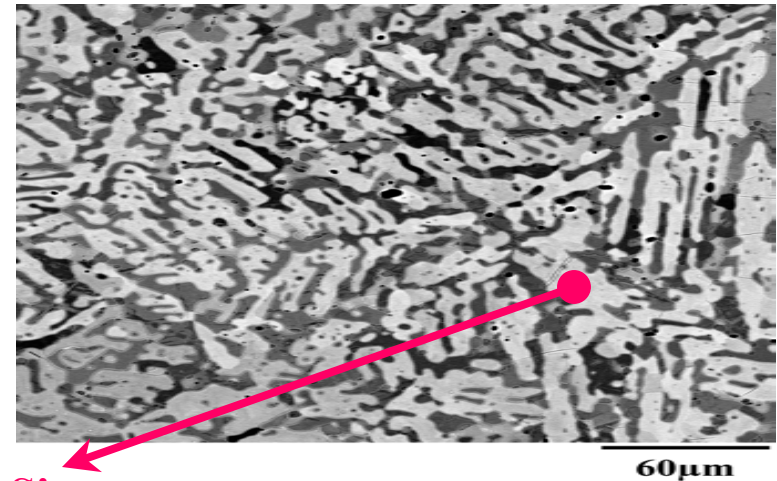
Nb-14Si-12.5Ti-10Cr



Nb_5Si_3
Nb-14Si-12.5Ti-10Cr-10Al

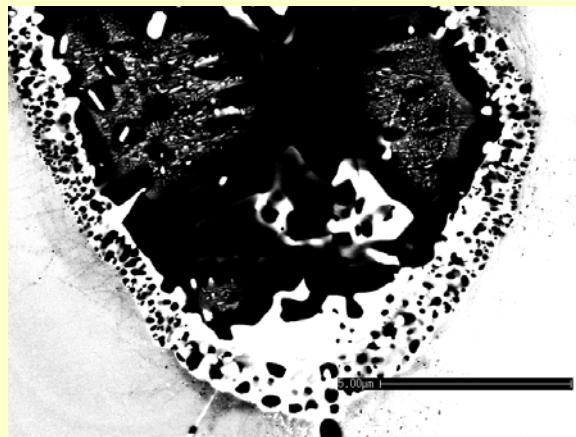
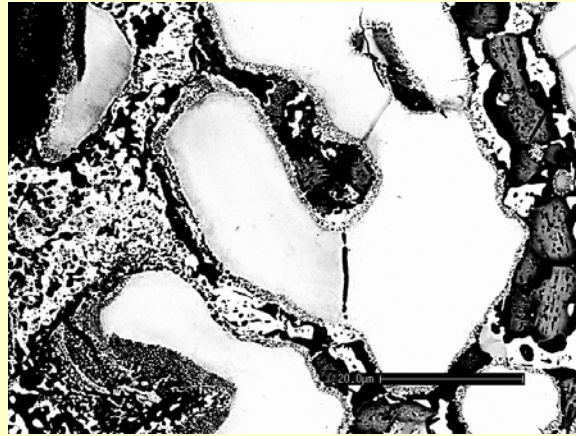


Nb-12.5Ti-14Si-10Mo-10Al
-5Cr-5Hf-5Zr



Nb_5Si_3
Nb-12.5Ti-14Si-20Mo-10Al
-9Cr-5Hf-5Zr

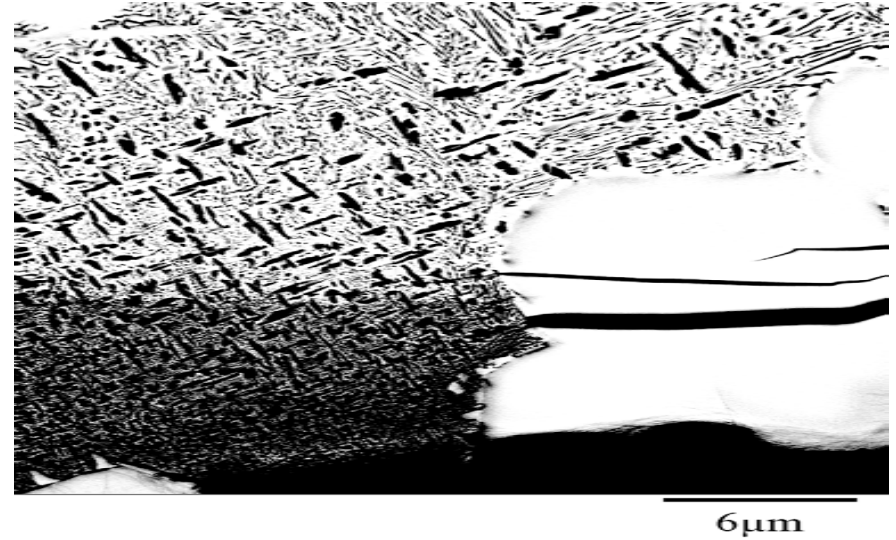
**Preferential oxidation of phases in
Nb-20Ti-20Si-4Ge-10Cr-3Al-4Hf-3B
Oxidized at 1200°C for 48 hrs**



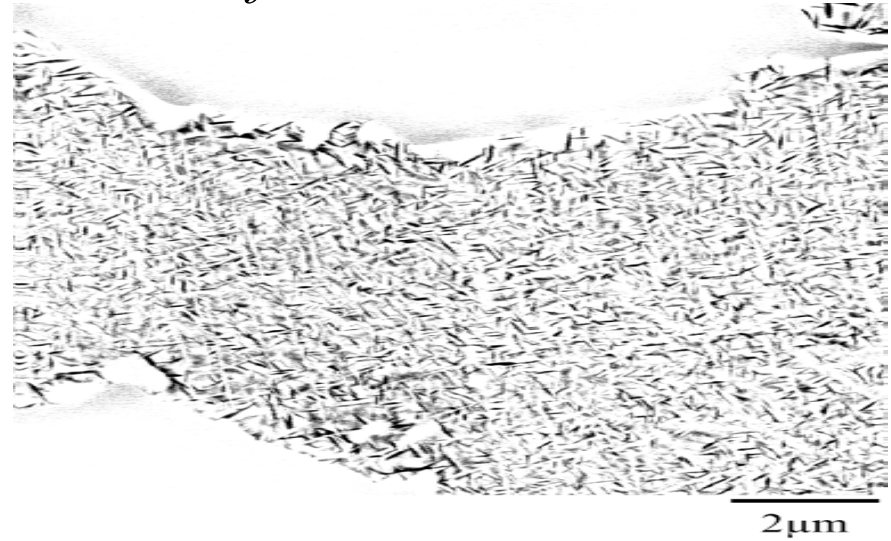
Oxidation of Nb-30Ti-7Si-10.5Cr-9.5Al -1.1Hf-1.5Zr-0.08C



1000 °C for 24 hours

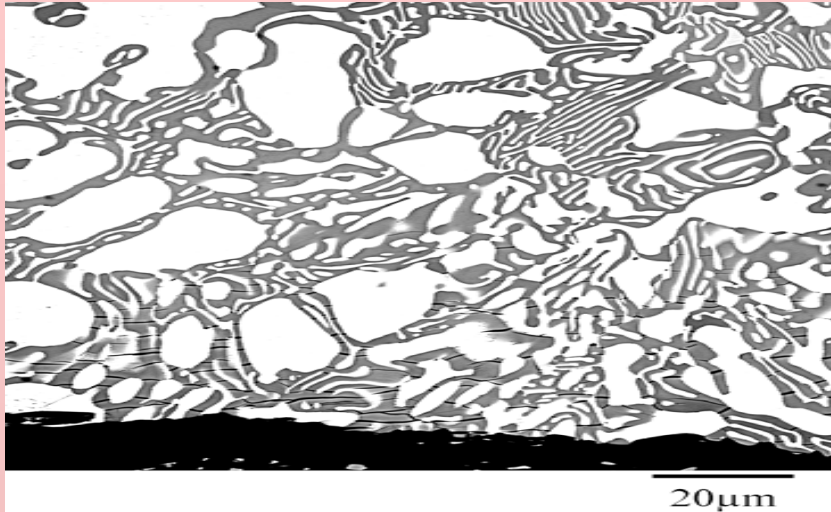


900 °C for 16 hours

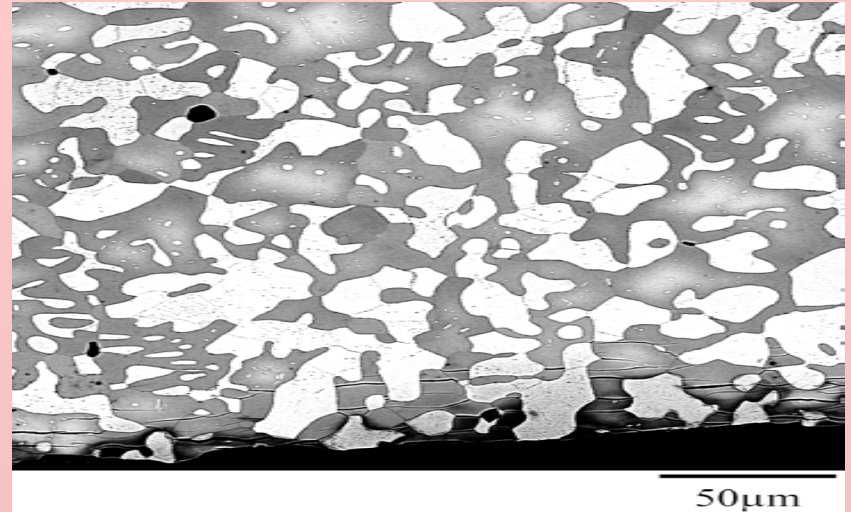


Oxidation of Nb_3Si and lamellar $\beta + \text{Nb}_5\text{Si}_3$

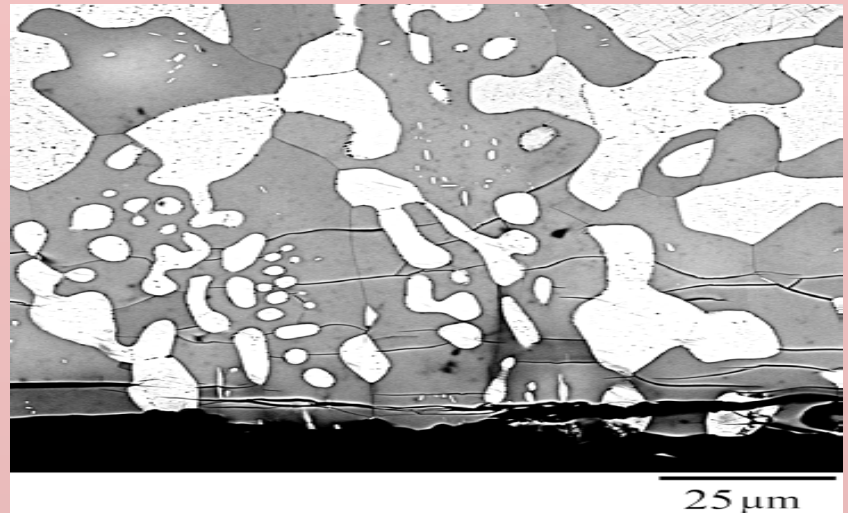
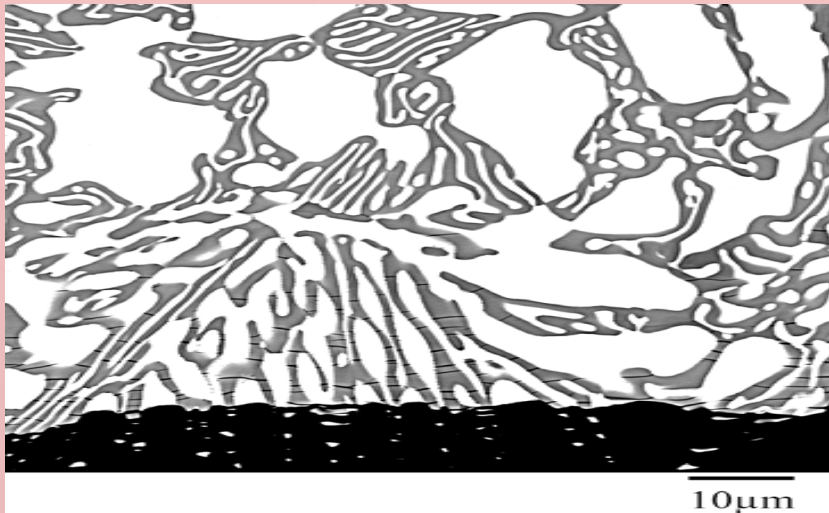
Nb-10.5Ti-16.5Si



Nb-20.8Ti-15.8Si



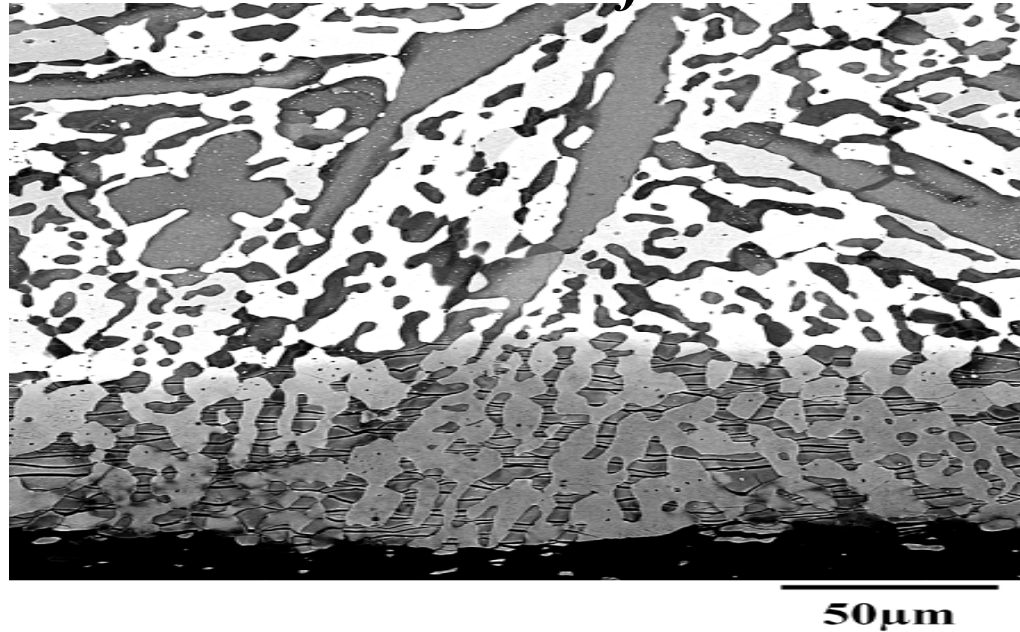
Oxidized at 1000 °C for 1 hr



Oxidized at 800 °C for 4 hrs

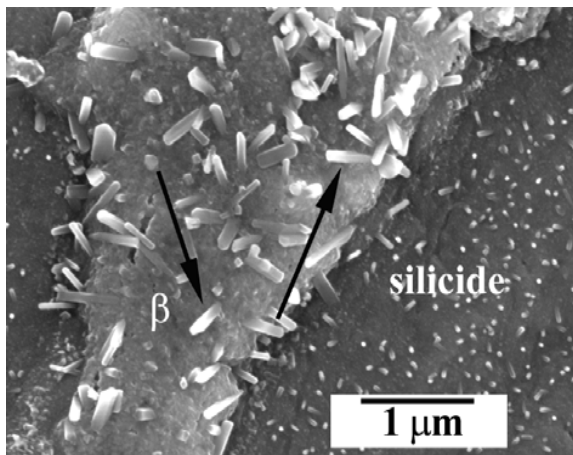
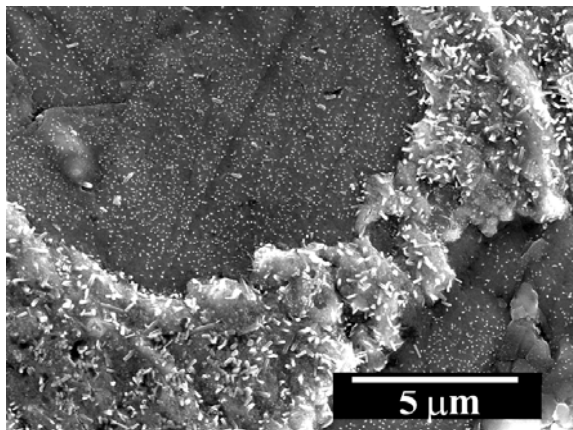
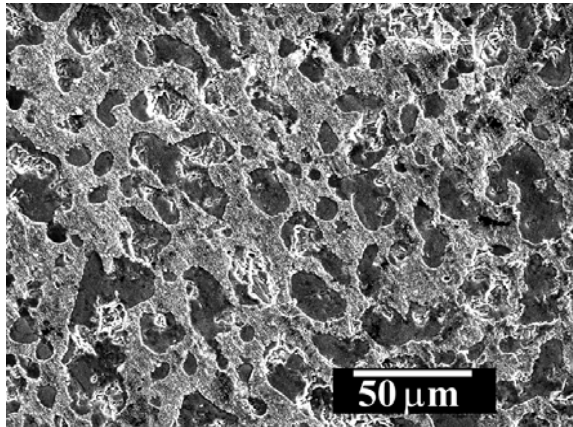


Nb-16.1Si-4.9Al 1 1000 °C for 1hr

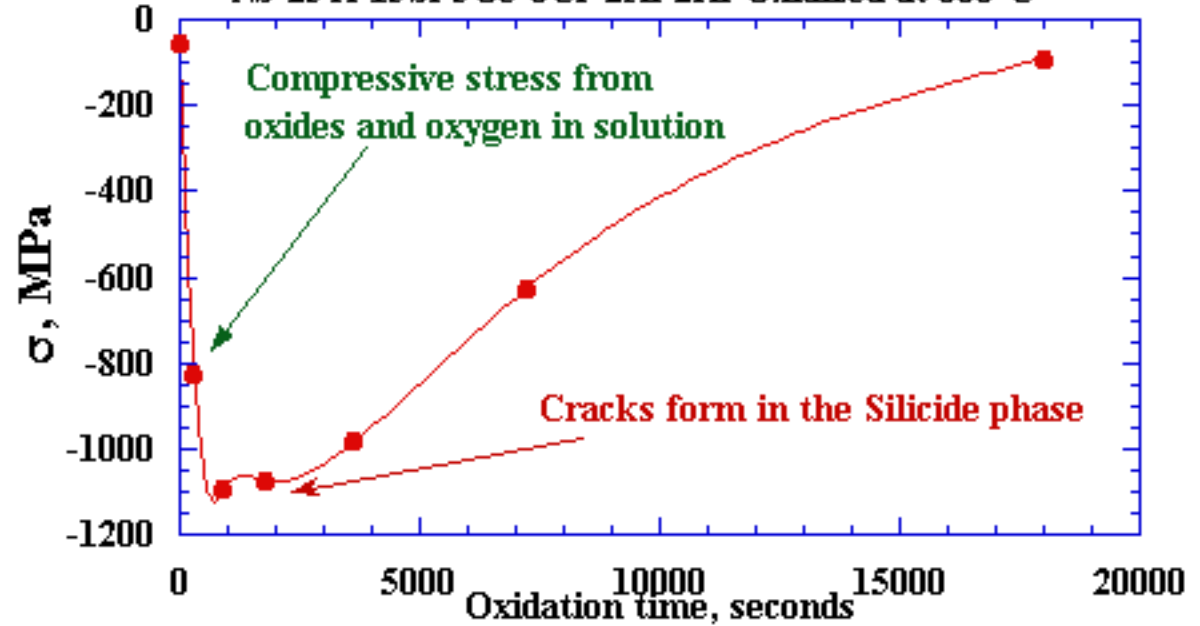


Nb-20.8Ti-15.7Si-4.3Al 800 °C for 4hrs

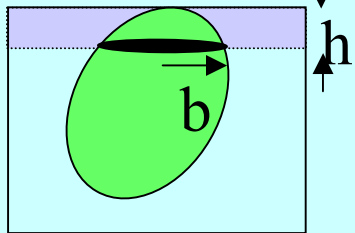
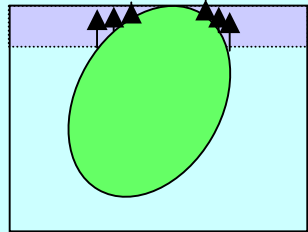
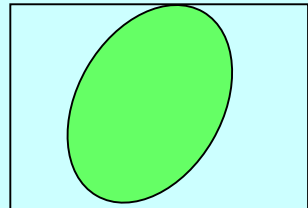
**Growth of surface oxide in
Nb-26Ti-13Si-5Ge-7Cr-2Al-2Hf-0.5Sn
alloy after oxidation at 800°C/ 5 hrs.**



Residual stresses as determined by XRD in
Nb-25Ti-13Si-5Ge-6Cr-2Al-2Hf Oxidized at 800°C

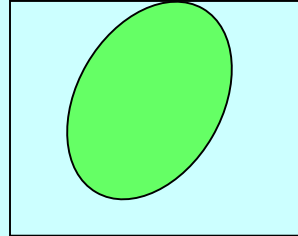


Mechanism of low temperature cracking



$$\tau = 0.5 (b/h)(\sigma_f)$$

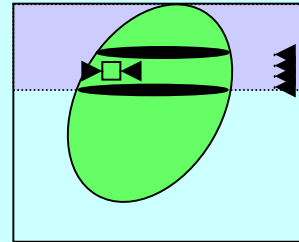
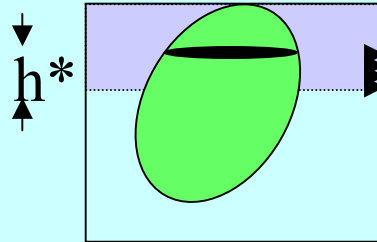
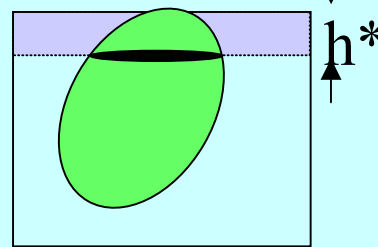
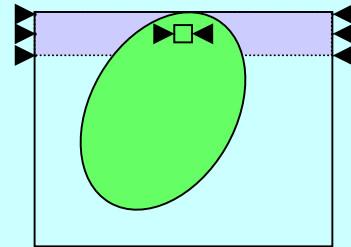
$(b \sim 5h \Rightarrow \tau \text{ high})$



$$E=200 \text{ GPa}$$

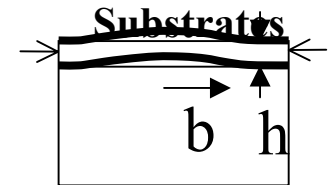
$$\nu=0.3 ; \phi=2$$

$$G=10 \text{ J/m}^2$$



decohesion/buckling releases stored strain energy at h^*

Compression Buckling of Thin Films on Substrates

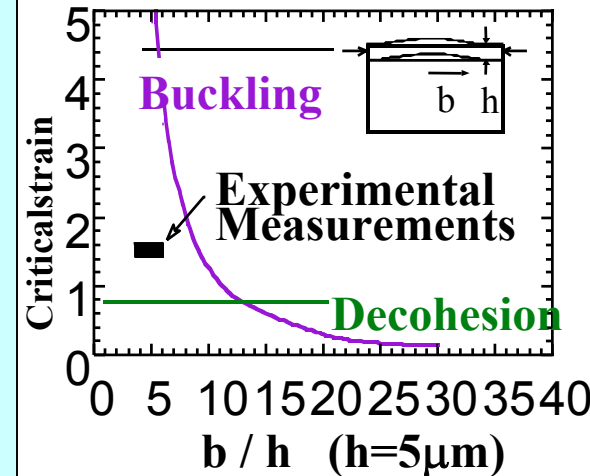


Energy Balance

$$\sigma_{\text{decoh}} = \phi \sqrt{\frac{E\Gamma_i}{(1-\nu)h}}$$

Buckling

$$\frac{b}{h} = 1.1 \sqrt{\frac{E}{\sigma_b}}$$



Conclusions

- It is possible to significantly modify the microstructural distribution of the phases in Nb alloys.
- Stability of Nb_3Si & Nb_5Si_3 is strongly influenced by alloying additions : Thermodynamic parameters associated with multicomponent systems must be modified.
- Oxidation resistance of Nb alloys can be increased by alloying.
- Oxidation behavior is affected by phase distribution in the material.
- It maybe possible to control the low temperature cracking by microstructural control.